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Ronald D. Archer, Valentino J. Tramontano, Ven O. Ochaya, Paul V. West & William Cumming

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Over the past few years we have developed a number of linear coordination polymers which have interesting properties. One type of study has centered on polymers which would provide sensitive lithographic resists based on the fact that the photoelectric effect interacts with nuclei approximately to the fourth power of the atomic number. We have prepared a number of uranyl carboxylate polymers which are very sensitive to gamma and electron beam radiation with α values higher than any currently available resist material [Archer, Hardiman, & Lee, 1987]. We have extended this work to cobalt and chromium coordination polymers as well. Coordination polymers of other metals are also under development.

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22a NAME OF RESPONSIBLE INDIVIDUAL
Dr. Kenneth Wynne22b TELEPHONE (Include Area Code)
(202) 9696-4409

22c OFFICE SYMBOL

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METAL COORDINATION POLYMERS AS POTENTIAL HIGH-ENERGY
LITHOGRAPHIC RESISTS

by

R. D. Archer, V. J. Tramontano, V. O. Ochaya,
P. V. West and W. Cumming

Department of Chemistry, University of Massachusetts
Amherst, Massachusetts 01003

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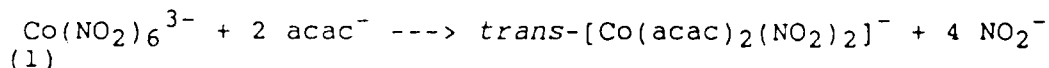
METAL COORDINATION POLYMERS AS POTENTIAL HIGH-ENERGY LITHOGRAPHIC RESISTS

R. D. Archer, V. J. Tramontano, V. O. Ochaya,
P. V. West, and W. Cumming

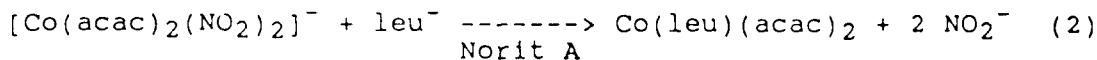
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INTRODUCTION

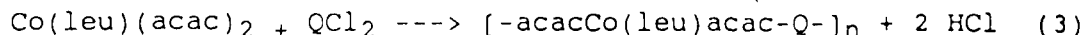
High energy radiation can modify polymers by causing cross-linking and/or degradation. One particularly interesting application is the use of polymers as photo- or electron-resists in lithography (1). Higher density integrated circuit chips have allowed metal oxide semiconductor random access memory device capacity to increase by several orders of magnitude in the recent past with minimum feature size of less than one micron at present. Further reductions are in progress, but diffraction effects will eventually limit the resolution of visible and ultraviolet sensitive resists. However, higher energy electron beams, X-ray, gamma ray, and ion-beam systems can provide the necessary resolution required for submicron resolution. But organic polymers have limited sensitivity to higher energy radiation with G values (chemical events/100 eV absorbed) typically not much greater than one, though sulfur containing organic polymers with G values of about 10 are known. We are using a combination of high G value polymers together with heavy metal atoms (which provide an increased absorption of energy through the photoelectric effect's approximately Z^4 power dependence, where Z = atomic number (2), Figure 1) in order to develop polymers which are very sensitive to high energy radiation (3,4). Whereas the advantage of heavy metal atoms is quite apparent from the uranyl species which we have studied--G_s values of 50 and up even at 662 keV, in the usual 10 - 20 keV range of electron beam studies, 3d-transition-metal polymers should have a tremendous advantage over simple organic polymers. Therefore, in order to get the advantage of the heavier atoms together with the sensitivity of the organic polymers containing sulfide and sulfone bridges, we have synthesized several cobalt(III) coordination polymers, one of which was briefly described earlier (5,6). The general synthesis for three polymers derived from leucinatobis(2,4-pentanedionato)cobalt(III), Co(leu)(acac)₂, is shown in equations 1 to 3.



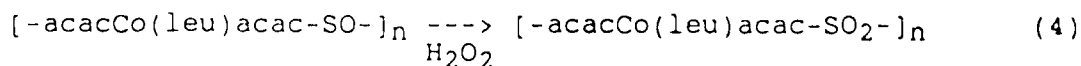
where acac⁻ is the anion of 2,4-pentanedione,

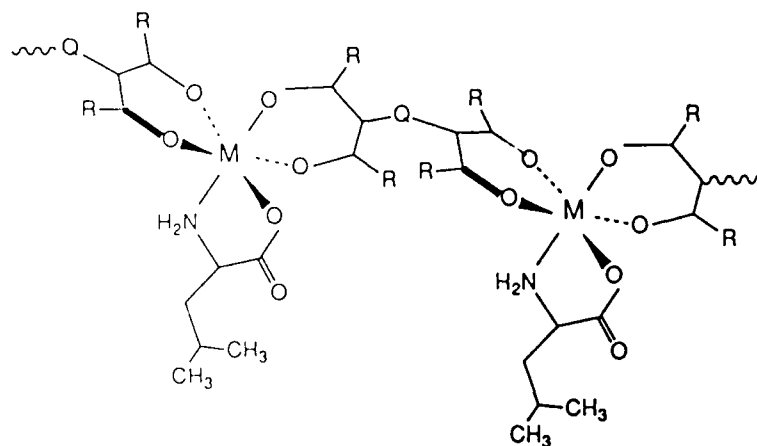


where leu⁻ is the anion of leucine,



where Q = S, S₂, or SO, and bridges the beta-diketones from adjacent units in the polymer chains. The corresponding sulfone polymer can be synthesized from the oxidation of the sulfoxide polymer with hydrogen peroxide.





Part of typical polymer chain, Q = S, SS, SO, SO₂, M = Co, R = CH₃

As noted below, the cobalt(III) coordination polymers have excellent G_s values, where G_s is the G value for scission. On the other hand, they appear to undergo cleavage only at the C-S and S-S bonds when irradiated at high energy, rather than at the cobalt centers [which would be expected in the ultraviolet region, where cobalt(III) to cobalt(II) charge transfer chemistry occurs for cobalt(III) beta-diketone complexes (7)]. In fact, other than the sulfoxo species, they all appear to have G_s values at least as high as any organic polymers. Coupled with the higher amount of energy absorbed for identical films and their good adhesion to silica-coated silicon, these species have excellent resist potential. In response to the concern of resist specialists about residual metal ions, it is gratifying to note that no detectable free cobalt ions can be observed after several megarads of irradiation at high energies, either for the monomer or the polymer. This observation led us to consider the more volatile fluorinated chromium(III) beta-diketonates. Progress with the chromium species is also noted herein, though this work has not proceeded as far as the cobalt(III) studies.

EXPERIMENTAL

Solvents and ligands were carefully dried prior to use. Sulfur dichloride and sulfur monochloride were purified just prior to use by appropriate literature methods (8,9).

(S-Leucinato-*N,O*)bis(2,4-pentanedionato-*O,O'*)cobalt(III).--This neutral complex was prepared by the method of Laurie (10). Yields of 20 - 29% of pure Co(leu)(acac)₂ were obtained from the reaction between Na[*trans*-(acac)₂(NO₂)] and Na[S-(leu)] after column chromatography, rotary evaporation and recrystallization.

Anal. Calcd for C₁₆H₂₆NO₆Co: C, 49.6; H, 6.8; N, 3.6.
Found: C, 49.5; H, 6.7; N, 3.6.

(S-Leucinato-*N,O*)bis[2,2,6,6-tetramethyl-3,5-heptanedionato-*O,O'*]cobalt(III).--An analogous reaction between the corresponding 2,2,6,6-tetramethylheptanedionate (or dipivaloylmethanate, dpm), but at much lower concentrations due to limited solubility in water, provided a 15% yield of the intermediate product and a 25% yield of the final Co(leu)(dpm)₂ product. FT-NMR carbon-13 results are in agreement with the formulation given.

Anal. Calcd for C₂₈H₅₀NO₆Co: C, 60.5; H, 9.1; N, 2.5.
Found: C, 60.3; H, 9.0; N, 2.5.

Poly((S-leucinato-*N,O*)-u-[3,3'-dithiobis(2,4-pentanedionato-*O,O'*)]cobalt(III)).--The dropwise addition of 1.7460 g disulfur dichloride (12.93 mmol) in 6 mL dry dichloroethane to 5.0088 g Co(leu)(acac)₂ in 20 mL dimethylacetamide (DMAC) with 0.7095 g sodium carbonate (6.69 mmol) as a slurry in the dimethylacetamide solution under argon and with vigorous stirring. The mixture was stirred for 24 - 36 hr and then precipitated with diethyl ether. Alternatively, the solvent was

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removed *in vacuo* at 45°C. The product was collected on a fritted funnel, washed with water, and dried *in vacuo* at 100°C; yield, 5.5 g; 95%. The polymer was fractionated (three times) by a DMAC/acetone or di-methyl sulfoxide/acetone solvent/nonsolvent precipitation to re-move low molecular-weight material.

Anal. Calcd for $[C_{16}H_{24}NO_6S_2Co]_n$: C, 42.8; H, 5.4; N, 3.1; S, 14.3. Found: C, 42.5; H, 5.1; N, 2.9; S, 14.2.

Poly((S-leucinato-*N,O*)-u-[3,3'-thiobis(2,4-pentanedionato-*O,O'*)]cobalt(III)).--The dropwise addition of 0.7592 g sulfur dichloride (7.37 mmol) in 6 mL dry dichloroethane to 2.8514 g $Co(acac)_2(leu)$ (7.36 mmol) in 60 mL dichloroethane or dichloro-methane under argon with vigorous stirring. After 24 hr of stirring HCl and the solvent were removed *in vacuo*, washed with water, and dried *in vacuo* at 100°C; yield, 2.76 g; 90%. The polymer was fractionated by an acetone/water fractional precipitation.

Anal. Calcd. for $[C_{16}H_{24}NO_6SCo]_n$: C, 46.0; H, 5.8; N, 3.4; S, 7.7. Found (from $C_2H_4Cl_2$): C, 45.4; H, 5.7; N, 3.0; S, 7.3. Found (from CH_2Cl_2): C, 45.9; H, 5.6; N, 2.8; S, 7.5.

(The use of more polar solvents such as acetonitrile with an acid acceptor proved less satisfactory with SCl_2 than with S_2Cl_2 above.)

Poly(S-leucinato-*N,O*)-u-[3,3'-sulfoxobis(2,4-pentanedionato-*O,O'*)]cobalt(III)).--The dropwise addition of 0.9213 g thionyl chloride (7.74 mmol) in 4 mL dry dichloroethane to 2.9925 g $Co(acac)_2(leu)$ in 10 mL dimethylformamide and 15 mL dichloroethane under argon with vigorous stirring at 0°C. The mixture was then allowed to stir for at least 24 hr. The solvent and any residual HCl was removed at 45°C *in vacuo*, and the product was dried at 100°C; yield, 3.13 g; 93%. The polymer was fractionated with an acetone/water fractional precipitation.

Anal. Calcd for $[C_{16}H_{24}NO_7SCo]_n$: C, 44.3; H, 5.6; N, 3.2; S, 7.4. Found: C, 44.1; H, 5.3; N, 3.8; S, 6.9.

Poly(S-leucinato-*N,O*)-u-[3,3'-sulfonebis(2,4-pentanedionato-*O,O'*)]cobalt(III)).--The dropwise addition of 0.10 mL hydrogen peroxide to 0.0603 g fractionated sulfoxide polymer in 10 mL acetonitrile with vigorous stirring. The mixture was heated to 40°C and stirred for 24 hr. The solvent was removed *in vacuo* at 40°C, and the sample was then dried *in vacuo* at 100°C.

Anal. Calcd for $[C_{16}H_{24}NO_8SCo]_n$: C, 42.8; H, 5.4; N, 3.1; S, 7.1. Found: C, 41.0; H, 5.6; N, 3.4; S, 6.9.

Poly((111-trifluoro-2,4-pentanedionato-*O,O'*)chromium(III)-u-[3,3'-thiobis(2,4-pentanedionato-*O,O'*)] $Cr(tfa)(acac)_2S$).--The synthesis of this polymer has been based on the reaction of $Cr(tfa)(acac)_2$, which was prepared by the method of Palmer, Fay and Piper (11) with SCl_2 (exact 1:1 mole ratio) in methylene chloride at -10°C. The infrared spectra and the intrinsic viscosity of 0.17 dL/g for the product are consistent with polymerization though the GPC results were less conclusive.

Physical Characterizations.--Gel permeation chromatography, viscosity, scribe-stripping, nuclear magnetic resonance, infrared, and ultraviolet-visible measurements were performed as reported earlier (12). Electron spin resonance spectroscopy measurements were made with an IBM ESP 300 instrument with quartz tubes sealed *in vacuo* prior to irradiation. To remove color centers from the quartz, one end of the tube was heated with a torch while the other was immersed in liquid nitrogen. The sample was then reversed and the process repeated.

Polymer Irradiation.--Samples of the polymers sealed *in vacuo* were exposed to cesium-137 gamma irradiation (662 KeV) at doses ranging from 0.02 to 0.07 Mrad/hr.

RESULTS AND DISCUSSION

Linear coordination polymers of cobalt(III) have been prepared from the reactions of $\text{Co}(\text{leu})(\text{acac})_2$ with SCl_2 , S_2Cl_2 , and SOCl_2 . The solvents chosen for the different reactions have been determined from a knowledge of the reactivity of the sulfur halides, from the need for solubility throughout the polymerization reaction, and by trial and error. The sulfone polymer was prepared by hydrogen peroxide oxidation of the sulfoxide polymer. Several properties of the polymers are provided in Table 1.

Table 1. Linear cobalt(III) polymers derived from $\text{Co}(\text{leu})(\text{acac})_2$

Bridge ^a	$M_n(\text{GPC})^b$	$M_n(\text{NMR})^c$	Visc ^d	UV/Vis ^e	TGA ^f	Infrared ^g
-S-S-	34,000	$\geq 13,000$	0.11	280/553	180°	h,1
-S-	13,500	$\geq 15,000$	0.10	279/546	160°	h,1
-SO-	16,000	-	-	276/553	-	1025 ^{h,1}
-SO ₂ -	17,000	$\geq 10,000$	0.084	279/548	190°	1160 ^{h,1}

^areplaces gamma hydrogens of acac's; ^bNMP solvent; polystyrene standards; ^cbased on FT-NMR of weak gamma proton signal in DMSO-d₆; ^dintrinsic viscosity, dL/g in NMP at 30°C; ^enm, molar extinction coefficients about 10^4 and $10^2 \text{ M}^{-1}\text{cm}^{-1}$ per repeating unit; ^ftemperature of first major weight loss; ^g cm^{-1} ; ^h780 cm^{-1} monomer gamma C-H mode not observed; ⁱ1550 cm^{-1} peak replaces two peaks observed at 1570 and 1520 cm^{-1} in monomer.

The number-average molecular weights may be higher than the NMR values because the gamma proton signals (one proton per chain is expected) are still at the noise level after several thousand transients. However, the viscosity of the dithio polymer is more like that expected for the NMR molecular weight value than for the GPC one.

The irradiation of these cobalt polymers with 662 keV gamma rays provides approximate G_S values of 18, 10, 0.4, and 60 for the dithio, thio, sulfoxo, and sulfone polymers, respectively, when Charlesby's equation (13) is used along with GPC molecular weight results. Whereas these values might seem quite high, polybutylene sulfone (PBS) has been reported to have G_S values of 12.2 and 23.7 *in vacuo* and in air, respectively (14). However, since the mass absorption coefficient for the more massive cobalt unit is 3.6 times that of PBS, the value we have obtained is reasonable. The electron spin resonance spectra of the irradiated polymers suggest radicals centered primarily on the sulfur atoms, with about 5% delocalization to the cobalt centers based on the hyperfine structure of the dithio and thio polymers. No hyperfine splitting is observed for the sulfone polymer, and the monomer shows no ESR signal at all. This verifies the observation that no spectral change is observed for extensive cobalt-60 irradiation of the monomer in methanol solutions. Evidence for SH peaks about 2500 cm^{-1} has been obtained by Fourier transform infrared spectroscopy after irradiation. Also, a loss in the intensity of the symmetrical SO₂ stretch near 1160 cm^{-1} after irradiation has been observed for the sulfone polymer. These results coupled with our ability to make films of the dithio polymer on silicon wafers which have oxidized surfaces looks encouraging. Unfortunately, the films peel from the surface when treated with the solvent combinations we have used in an attempt to determine the sensitivity to electron beams.

We have also expanded our studies to include chromium(III) systems. However, for the chromium centers we have chosen the more volatile trifluoro (tfa⁻) and hexafluoro (hfa⁻) derivatives of 2,4-pentanedione. Since the fluorinated derivatives are deactivated relative to the electrophilic attack used in these polymerizations, the $\text{Cr}(\text{acac})_2(\text{tfa})$ mixed ligand complex has been used initially to obtain polymeric products with sulfur halides.

We have also nitrated the $\text{Cr}(\text{acac})_2\text{-(tfa)}$ complex and reduced the nitro groups to amines to provide links with organic oligomers. Unfortunately, the first nitro together with the trifluoro group deactivates the system such that no dinitro product can be obtained.

- SUMMARY

Four cobalt polymers have been synthesized, three of which are very sensitive to gamma irradiation with resulting G_s values which exceed any known organic polymers. Furthermore, the cobalt is not released during the scission reactions. Films which adhere well enough for commercial use have not yet been obtained for the cobalt systems.

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Dr. Harry R. Allcock
Department of Chemistry
Pennsylvania State University
University Park, PA 16802

4132007

Dr. Ronald D. Archer
Department of Chemistry
University of Massachusetts
Amherst, MA 01003

413c028

Dr. William J. Bailey
Department of Chemistry
University of Maryland
College Park, MD 20742

413a006

Dr. Frank D. Blum
Department of Chemistry
University of Missouri - Rolla
Rolla, MO 65401

413m005

Dr. F. James Boerio
Materials Science & Engineering Dept.
University of Cincinnati
Cincinnati, Ohio 45221

413m012

Dr. R. S. Atkinson
School of Chemical Engineering
Georgia Institute of Technology
Atlanta, GA 30332

4132033

Dr. Chris W. Allen
Department of Chemistry
University of Vermont
Burlington, VT 05405

413c012

Dr. Ali S. Argon
Mechanical Engineering Department
Massachusetts Institute of Technology
Cambridge, MA 02139

a400005df

Dr. Kurt Baum
Fluorochem, Inc.
680 S. Ayon Avenue
Azusa, CA 91702

4000021sbi

Dr. Len J. Buckley
Naval Air Development Center
Code 6063
Warminster, PA 18974

Dr. Ivan Caplan
DTNSRDC Annapolis
Code 0125
Annapolis, MD 21401

Dr. Robert E. Cohen
Department of Chemical Engineering
Massachusetts Institute of Technology
Cambridge, MA 02139

4132001

Dr. E. Fischer
DTNSRDC Code 2853
Annapolis, MD 21402

413h006

Dr. Curtis W. Frank
Department of Chemical Engineering
Stanford University
Stanford, CA 94305

413h005

Dr. Gregory S. Girolami
School of Chemical Sciences
University of Illinois
Urbana-Champaign, IL 61801

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Department of Chemistry
California Institute of Technology
Pasadena, CA 91124

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Texas A&M University
College Station, TX 77843

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Department of Chemical Engineering
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Madison, WI 53706

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Dr. Warren T. Ford
Department of Chemistry
Oklahoma State University
Stillwater, OK 74078

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Dr. John K. Gillham
Department of Chemical Engineering
Princeton University
Princeton, New Jersey 08544

413c005

Dr. Eernard Gordon
Department of Polymer Science
Pennsylvania State University
University Park, PA 16802

413c025

Dr. Henry K. Hall
Department of Chemistry
University of Arizona
Tucson, AZ 85721

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Department of Physics
University of California, Santa Barbara
Santa Barbara, CA 93106

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Dr. Pat J. Hendra Department of Chemistry University of Southampton Highfield Southampton SO9 5NH United Kingdom 4134001	Dr. Charles E. Hoyle Department of Polymer Science University of Southern Mississippi Hattiesburg, MS 39406-0076 413c026	Dr. Joseph H. Magill Materials Science & Engineering Dept. University of Pittsburgh Pittsburgh, PA 15161 413c013	Dr. Leo Mandelkern Department of Chemistry Florida State University Tallahassee, FL 32306-3015 4132018
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Dr. Arnold Riser
Institute of Imaging Sciences
Polytechnic University
333 Jay Street
Brooklyn, NY 11201

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Dr. Charles M. Roland
Code 6120
Naval Research Laboratory
Washington, DC 20375-5000

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Dr. Ronald Salovey
Department of Chemical Engineering
University of Southern California
Los Angeles, CA 90089

413m010

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Dept. of Mechanics & Materials Science
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Cambridge, MA 02139

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Pittsburgh, PA 15213

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Department of Chemistry
Massachusetts Institute of Technology
Cambridge, MA 02139

4132038

Dr. David S. Soane
Department of Chemical Engineering
University of California, Berkeley
Berkeley, CA 94720-9989

413h004

Dr. Les H. Sperling
Materials Research Center #32
Lehigh University
Bethlehem, PA 18015

413c002

Dr. C. S. Sung
Institute of Materials Science
University of Connecticut
Storrs, CT 06268

413m011

Dr. C. H. Wang
Department of Chemistry
University of Utah
Salt Lake City, Utah 84112

413c020

Dr. Robert A. Weiss
Department of Chemical Engineering
University of Connecticut
Storrs, CT 06268

a400006df

Dr. Garth L. Wilkes
Department of Chemical Engineering
Virginia Polytechnic Institute
Blacksburg, VA 24061

4132020

Dr. Richard S. Stein
Polymer Research Institute
University of Massachusetts
Amherst, MA 01002

4132008

Dr. Sukant K. Tripathy
Department of Chemistry
University of Lowell
Lowell, MA 01854

4132016

Dr. Kenneth B. Wagener
Department of Chemistry
University of Florida
Gainesville, FL 32611

a400007df

Dr. George M. Whitesides
Department of Chemistry
Harvard University
Cambridge, MA 02138

4132010